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AFCRL-TR-75-0582
PHYSICAL SCIENCES RESEARCH PAPERS, NO. 650

Vacuum Hot-Pressing Apparatus and Techniques

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11 November 1975

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SOLID STATE SCIENCES LABORATORY PROJECT 5620
AIR FORCE CAMBRIDGE RESEARCH LABORATORIES
HANSCOM AFB, MASSACHUSETTS 01731

AIR FORCE SYSTEMS COMMAND, USAF

ACCESSION for

NTIS White Section
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Dist. AVAIL. and or

Unclassified

SECURITY CLASSIFICATION OF THIS PAGE (When Data Entered)

REPORT DOCUMENTATION PAGE

READ INSTRUCTIONS BEFORE COMPLETING FORM

1 REPORT NUMBER: AFCRL-TR-75-0582 AFCRL-PSRP-650

2 GOVT ACCESSION NO.

3 RECIPIENT'S CATALOG NUMBER

4 TITLE (and Subtitle): VACUUM HOT-PRESSING APPARATUS AND TECHNIQUES

5 TYPE OF REPORT & PERIOD COVERED: Scientific. Interim.

6 PERFORMING ORG. REPORT NUMBER: PSRP No. 650

7 AUTHOR(s): Joseph A. Adamski, Normantas Klausutis, ~~Major, USAF~~

8 CONTRACT OR GRANT NUMBER(s)

9 PERFORMING ORGANIZATION NAME AND ADDRESS:
Air Force Cambridge Research Laboratories (LQ)
Hanscom AFB
Massachusetts 01731

10 PROGRAM ELEMENT, PROJECT, TASK AREA & WORK UNIT NUMBERS: 562

11 CONTROLLING OFFICE NAME AND ADDRESS:
Air Force Cambridge Research Laboratories (LQ)
Hanscom AFB
Massachusetts 01731

12 REPORT DATE: 11 November 1975

13 NUMBER OF PAGES: 30

14 MONITORING AGENCY NAME & ADDRESS (if different from Controlling Office)

15 SECURITY CLASS (of this report): Unclassified

15a DECLASSIFICATION/DOWNGRADING SCHEDULE

16 DISTRIBUTION STATEMENT (of this Report):
Approved for public release; distribution unlimited.

17 DISTRIBUTION STATEMENT (of the abstract entered in Block 20, if different from Report)

18 SUPPLEMENTARY NOTES:
This work was carried out at the request of and with the financial support of AFWL.

19 KEY WORDS (Continue on reverse side if necessary and identify by block number):
Hot-forging
Hot-pressing
Alkali halides
Sodium chloride
Potassium chloride
Mechanical strength

20 ABSTRACT (Continue on reverse side if necessary and identify by block number):
This report describes the construction and operation of a vacuum hot-pressing apparatus and details of a technique for hot-forging pure and doped alkali halide single crystals to produce high-strength IR windows without degrading optical absorption.

DD FORM 1 JAN 73 1473 EDITION OF 1 NOV 65 IS OBSOLETE

Unclassified

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Preface

The authors gratefully acknowledge the cooperation and contributions of their colleagues in this program. J.R. Weiner rendered extensive assistance in the design, technical evaluation, procurement, and construction of the apparatus. R.M. Hilton, J.J. Larkin, J.J. O'Connor, and J.R. Weiner, grew the single crystals. A.D. Key fabricated the forging blanks and polished the forged billets.

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1. INTRODUCTION

The AFCRL Solid State Sciences Laboratory has actively participated in the High-Power Infrared Laser Window program since 1 January 1971, at the request of the Air Force Weapons Laboratory (AFWL). Designated the LQ-10 program at AFCRL, the work here has been concentrated on various phases of material selection, crystal growth, and material fabrication, characterization, and evaluation. One of the problems addressed in the LQ-10 program has been that of increasing the mechanical strength of laser window materials without degrading other properties.

One way to increase material strength is by fully compacting polycrystalline material into a dense, fine-grained mass. This can be achieved by hot-pressing powders. Unfortunately, the large surface area of powders is subject to chemical contamination, and the consequent aggregation of impurities at grain boundaries in the hot-pressed material causes unacceptable absorption and scattering in the finished laser window.

An alternative approach is to begin with a single crystal and hot-forge the sample under a temperature and pressure sufficient to cause pressure-induced recrystallization (PIR). The grain boundaries of such materials are free of absorbing and/or scattering impurities. In addition to strengthening the material,

(Received for publication 7 November 1975)

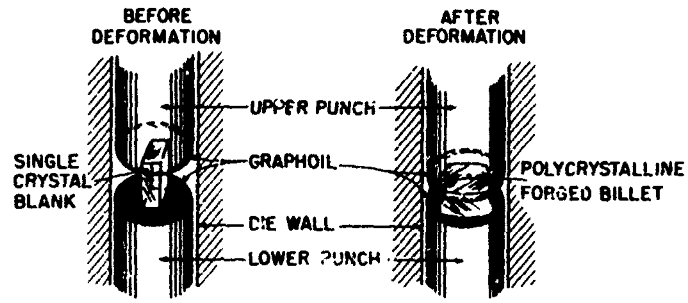


Figure 1. PIR Schematic Showing Fully Unconstrained Deformation

this fabrication technique (Figure 1) can readily be scaled to produce large-diameter windows.

At the time AFCRL was asked to undertake the LQ-10 program, the Laboratory had not had any real experience with the processes involved nor did it have any hot-forging apparatus. A preliminary study was made, and the basic design and specifications for a vacuum hot-pressing apparatus were drawn up. The system was engineered and constructed under contract awarded after competitive bidding. It was placed in operation on 8 May 1974. Experimental techniques were fully developed by September of 1974, and the first results on the RbCl-doped KCl system were presented in November of 1974.[1]

This report describes the salient features of the vacuum hot-pressing apparatus and the experimental techniques that have been successful. It is sufficiently detailed to serve as an instruction manual for users of this apparatus.

2. APPARATUS

The vacuum hot-pressing system shown in Figure 2 consists of several subsystems. Only those that are unique to the hot-pressing operation will be described in detail.

2.1 Vacuum Chamber

The vacuum chamber (Figure 3) is a horizontal cylinder 18 in. long, supported in a press frame. Its inside diameter is 24 in. The chamber and dished heads

1. Klausutis, N., Nikula, J., Adamski, J., Collins, C., Bruce, J., and O'Connor, J. (January 1975) Properties of hot-forged RbCl-KCl alloys of low rubidium concentrations, Proc. Fourth Annual Conference on Infrared Laser Window Materials, pp. 611-619. (Held in Tuscon, Ariz., 18 to 20 November 1974, sponsored by AFML of Wright-Patterson AFB); also AFCRL-TR-75-0179 (AD No. A008480)

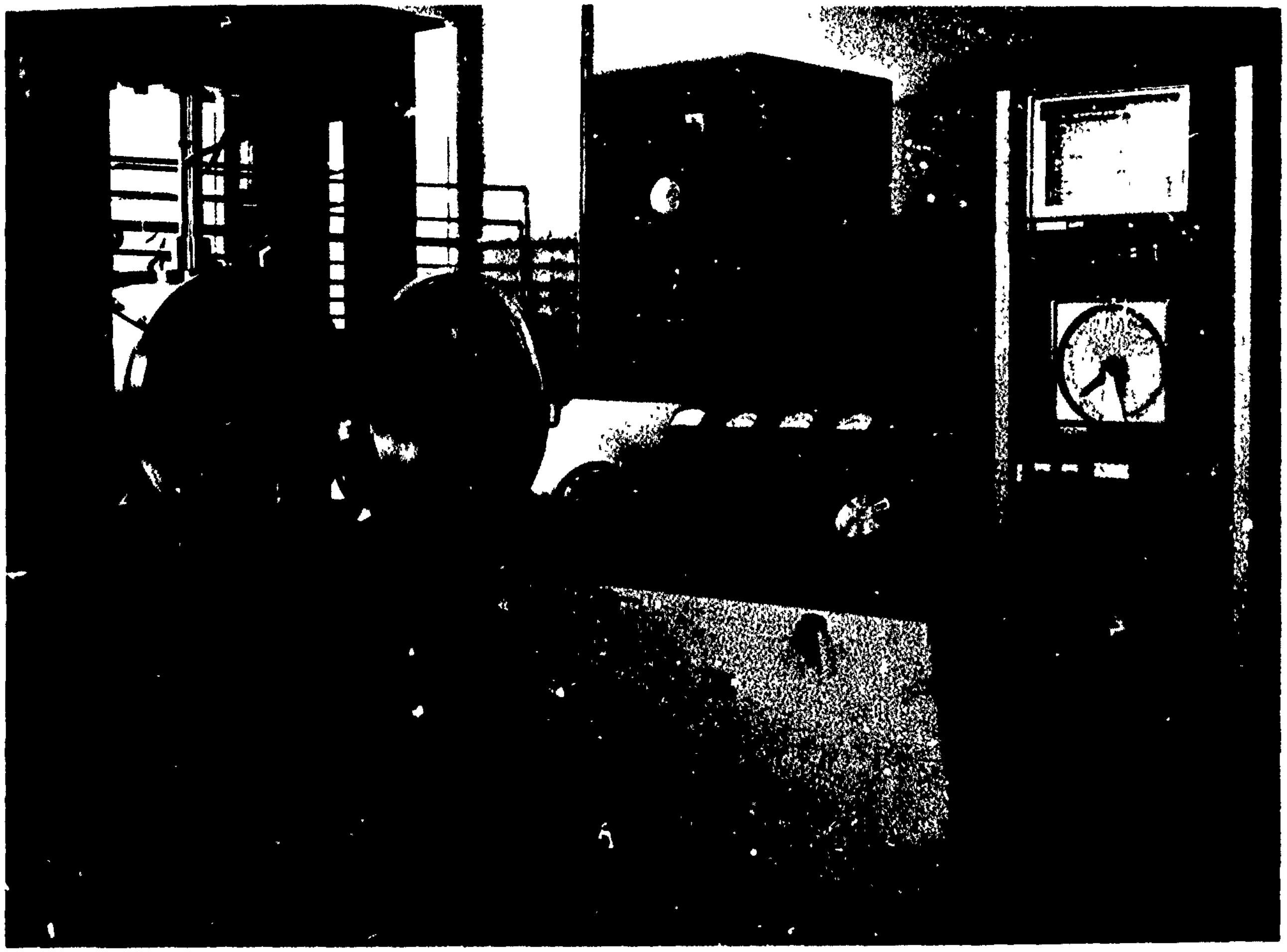
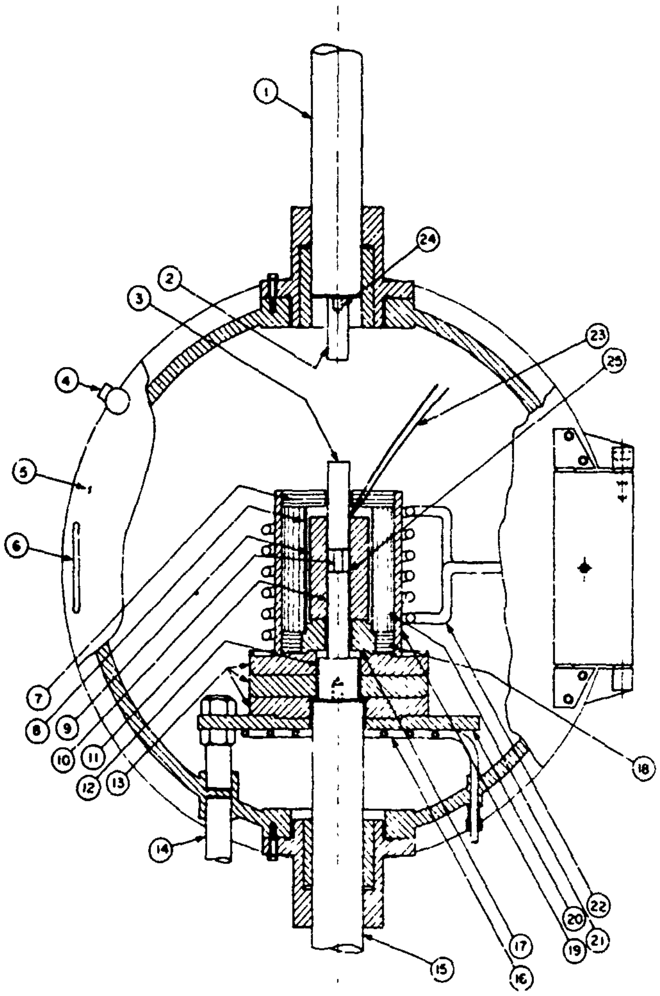


Figure 2. Vacuum Hot-Pressing System



1 UPPER RAM
2 RAM EXTENSION
3 UPPER PUNCH
4 SWING CLAMP
5 CHAMBER DOOR
6 HANDLE
7 GRAPHITE FELT INSULATION
8 GRAPHITE SHIELD
9 DIE BODY
10 CRYSTAL SAMPLE
11 LOWER PUNCH
12 RAM EXTENSION
13 DIE SUPPORT DISKS
14 DIE PLATFORM SUPPORT ROD
15 LOWER RAM
16 DIE PLATFORM COOLING
17 DIE MOUNTING BLOCK
18 GRAPHITE FELT INSULATION
19 QUARTZ TUBE
20 DIE PLATFORM
21 GRAPHITE FELT INSULATION
22 RF HEATING COIL
23 THERMOCOUPLE
24 THREADED STUD
25 GRAPHOIL

Figure 3. Vacuum Chamber Schematic

(back cover and front door) are completely water-jacketed. The outer and inner walls, dished heads, and all flanges, are made of 304 stainless steel. The front door on the chamber is hinged, opens fully, and can be held closed by four quick-acting clamps. An 8-in. power port provides for induction power feedthrough. Three sight ports—located in the top of the chamber, in the top of the door, and in the bottom of the door—provide views of the interior of the furnace chamber.

All three sight ports have swing shields mounted in front of the windows to prevent clouding during operation. The vacuum system is attached to a 6-in. pump port located at the rear dished head. A utility port accommodates a 1-in. flanged nozzle equipped with an octal header for the thermocouple feedthrough. There are also two 3-in. flanged ports, one in the top and one in the bottom of the vacuum chamber, with sliding seals for the rams. All connections for vacuum gages and vent and backfill valves are threaded.

2.2 Press Frame

The welded-steel press frame is of H-frame design, built to withstand a maximum working force of 60, 000 lb. It supports the hydraulic system, vacuum chamber, die platform, and vacuum-pumping system.

2.3 Hydraulic Power Supply and Control

The hydraulic power supply is a 10-hp unit supplying up to 5 gpm at 3100 lbf/in^2. It has a 35-gal oil reservoir, equipped with a temperature-indicating sight glass. The pump, a variable-displacement piston type, has pressure compensation at 3500 lbf/in^2. Its maximum capacity is 20 gpm. To prevent overloading the electric motor, its maximum volume is adjusted to approximately one-fourth its full stroke. Cooling water flows through a heat exchanger, regulated by a temperature-controlled valve that maintains the temperature of the oil in the reservoir.

The hydraulic equipment consists of two cylinders (bore: 5 in; stroke: 8-1/4 in.) front-flange-mounted in the press frame. At a pressure of 3056 lbf/in^2, these cylinders exert a maximum total force of 60, 000 lb.

The hydraulic supply has a regulating valve that can be manually adjusted from zero to 3100 lbf/in^2. Upper and lower vertical rams of 2-1/2-in-diam. stainless steel are attached to the cylinders. These rams are water-cooled and operate through sliding seals. They can be withdrawn to make the chamber available for general use.

The control console contains directional valves for manual and automatic control of the rams. Figure 4 is a schematic diagram of these hydraulic controls.

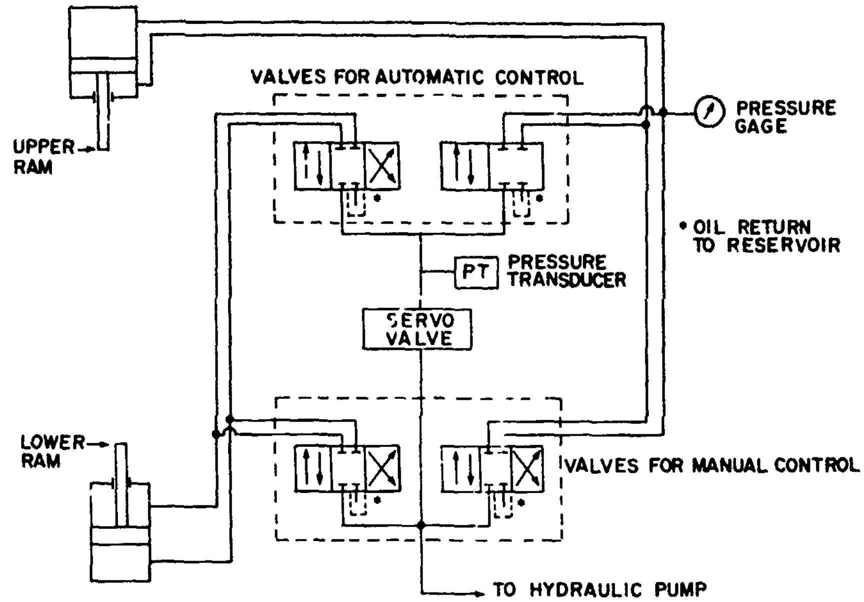


Figure 4. Hydraulic Control Schematic

The valves for automatic control—operating with the pressure transducer, servo valve, and pressure gage—maintain, record, and control a preset pressure to ±0.5%. The pressure is set on a four-digit Digivider* and responds to an electrohydraulic feedback control circuit. For extrusion or hot ejection the lower ram can be manually switched to move downward with the upper ram, which remains under automatic pressure control.

The valves for manual control, used primarily for positioning the rams, can raise or lower the upper and lower rams independently of each other. Flow restrictor valves inside the console are used for adjusting the upper limit of ram speed.

2.4 Vacuum System

The vacuum system has a 6-in. quick-acting main vacuum valve, a 6-in. diffusion pump whose capacity is 1500 ℓ/sec, a roughing pump, and foreline and roughing valves with a baffle/trap system.

*The Digitran Company, Pasadena, Calif.

2.5 Vacuum Instrumentation

The instrumentation is all solid-state. It has duel-thermocouple/single-station hot-filament-ionization gage control, complete with gage tubes and connection cable, for monitoring the vacuum over the range of 1000 μm to 2×10^{-9} torr. Complete fail-safe reliability is ensured through an adjustable overpressure protection circuit with bypass, resistance outgas circuit, and recorder output.

Vacuum interlocks are provided by separate contacts on the vacuum instrument. If pressure rises above full scale on a selected scale, the heat-zone contactor on the electrical power supply drops out, turning off the heat. When forgings are conducted in inert atmospheres, a bypass switch is used to defeat the vacuum interlock system.

Inert gas is introduced into the system through a manual needle valve to control the pressure from 1 to 5 lbf/in^{2}g. A compound Bourdon-tube gage, 5 lbf/in^{2}g, and pressure-relief valve are installed on the pumping port.

2.6 Automatic Temperature-Programing and Control

The automatic temperature recorder-controller is a Leeds and Northrup Speedomax H with a Model 80 Controller and a Leeds and Northrup Trend Trak Programer. The thermocouple used is tungsten—5% rhenium / tungsten—26% rhenium.

2.7 Electrical Power Supply

The power supply used is of the motor generator type, rated at 30 kW at a frequency of 4.2 kHz with unity power factor, and capable of producing temperatures up to 2000°C. It is horizontal, completely contained, and water-cooled to reduce windage noise and contamination by foreign matter.

The motor generator set has such features as a multitap water-cooled output transformer, an 8-position transformer tap switch, electrically operated capacitor contacts, and readouts of voltage, current, power, and power factor. A water-pressure switch is interlocked within the control console to ensure maximum generator protection.

2.8 Punches and Dies

The punch and die sets are 1- and 2-in. in diameter. Those used for forging at temperatures above 700°C are made of high-strength graphite (HPD-1)*; for temperatures below 700°C the sets are made of high-speed tool steel (AISI type M2).

The HPD-1 graphite is isotropic, homogeneous, extremely dense, of fine grain and high-tensile strength, and has a high coefficient of thermal expansion.

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*Poco Graphite, Inc., Decatur, Texas 76234

With pore diameters averaging 0.4 μm, it is also easily machinable to tolerances never before practical with graphite. These properties combine to make it unique among graphites and ideal for hot-pressing.

The steel sets work properly only when the top and bottom punches have a close sliding fit. The maximum clearance between the die body and the mating top and bottom punches must not exceed 0.0005 in. To achieve this, both punches and the hole in the die body must be honed and polished to a 12-microinch finish. The steel die sets are then heat-treated to Rockwell C58/60 hardness. The ends of the punches, honed and polished to an 8-microinch finish, must be flat and parallel to each other to 0.0002 in/in.

3. FORGING PROCEDURE

The procedure developed for forging KCℓ and KCℓ alloys will now be described in detail. By careful adjustment of the forging parameters—material composition, temperature, height reduction, and strain rate—it is possible to use this basic procedure for a wide variety of single crystals or single-crystal alloys.

3.1 Selection of Forging Parameters

A search of the literature discloses the parameters necessary for forging many common materials. Temperatures can vary over a considerable range. For example, some investigators have found that KCℓ can be forged at levels as low as 100°C; others have obtained satisfactory results at levels as high as 500°C.

Generally, other variables being held constant, lower temperatures produce small grain sizes whereas higher temperatures produce large grain sizes. At low temperatures, however, edge-cracking and tearing and veil formation may occur. These defects can be somewhat reduced by going to lower strain rates or higher temperatures. For KCℓ and KCℓ alloys we have found that strain rates in excess of 0.02 in/in/min produce unstable microstructures that can undergo grain growth at room temperature. Optimizing the parameters involves selecting a temperature high enough and a strain rate low enough to produce crack-free billets, at the same time keeping the temperature low enough to achieve small grain size. The amount of height reduction must be sufficient to ensure fully developed microstructures. (For further details see Appendix A.)

Optimum conditions can be determined by running three series of forgings. For the first, the temperature is varied while the percentage reduction and strain rate are kept constant; the grain sizes obtained are measured to determine the proper temperature for the desired size. In the second series, the selected temperature and the strain rate are kept constant while the percentage reduction

is varied; the resulting microstructures are examined, and the proper reduction for the desired microstructure is selected. In the third series of forgings, the only variable is the strain rate; after this run, the samples are examined to select the strain rate that minimizes cracking and edge-tearing.

The optimum hot-forging parameters—temperature, percentage reduction, and strain rate—that will produce the most nearly ideal billet have thus been determined. The method has been successfully used with single-crystal boules.

3.2 Preparation of Forging Blanks

Forging blanks can be fabricated from Czochralski or Bridgman boules. They need not be oriented except for the purpose of facilitating the correlation of data and prediction of how a sample will deform. With Bridgman boules, which are randomly oriented, the common practice is to forge along the axis of crystal growth and report the tilt angle, that is, the angle between the growth axis and nearest {100} plane.

To reduce the cost of crystal-growing and take advantage of the convenient shape of [100]-oriented halide boules, we used Czochralski air-grown boules for most of the work reported here. The boules were cut with a wet-string saw and/or water-polished to fit either the 1- or 2-in. die. The blanks used in our preliminary runs were generally rectangular, approximately 3/4 in. high by 1/2 in. to 3/4 in. square in cross section. For mechanical test bars we used forging blanks 1-1/2 in. to 2 in. high and 1-in. square in cross section.

Before each forging blank was loaded into the hot press it was subjected to a final inspection to ensure that it had no cracks, chips, or surface damage. It was then rinsed in distilled water and dried with Kimwipes to remove any sharp edges or small defects. Next, after its height, width, and depth were carefully measured, it was ready for loading. This procedure was followed for as-grown or annealed crystals.

3.3 Pressure-Transducer Calibration Procedure

The pressure transducer has a slight temperature-sensitivity. Because of the daily and seasonal temperature fluctuations in the laboratory, it may therefore be necessary to calibrate the pressure transducer before every forging run. There are nine steps in the procedure that we follow.

1) Turn off the hydraulic pressure supply. Reduce hydraulic pressure to zero gage pressure by turning the automatic control valves ON and OFF about 6 to 8 times (this allows the oil to bleed back to the hydraulic fluid reservoir).

2) Set the Digivider to zero.

3) Open the back of the hydraulic control console. Attach a precision voltmeter to Terminals No. 4 and Ground. (A resolution of 1 mV dc or better is required, and a digital voltmeter is recommended.)

[Note: It is recommended that the power ON-OFF switch for the hydraulic control system electronics be left in the ON position. This will save warm-up time and ensure thermal steady-state in the pressure transducer.]

4) With the pressure transducer sensing zero gage pressure, set the transducer output voltage as close to zero mV as possible.

[Adjust the transducer output slowly, turning the adjusting screw on the transducer nearest the electrical connection with a small screwdriver. If the correct polarity for all connections is observed, then an increase in pressure will cause an increase (positive) in the transducer output. Note, however, that when the transducer heats up, the output reading will indicate a pressure lower than actually present.]

5) Connect the precision voltmeter to the pressure signal terminals on the front of the hydraulic control console and set it on the mV scale. Adjust the trim potentiometer F (Board No. 2) so that the voltage reads slightly to the positive side of zero.

[Caution: Assume that both the transducer output and pressure signal are set exactly to zero, and that the automatic pressure selector switch is turned on. As soon as the hydraulic supply is turned on and the automatic control valves are set in the ON position, ram motion can begin even if the Digivider is still set on zero.

Defining the Digivider setting at which initial ram motion begins as the threshold, we have found it convenient to use a threshold between 10 and 20. Extremely low threshold values are undesirable because a temperature drift could actually produce ram action at thresholds below zero. This condition could result in a runaway, with uncontrolled forging.]

6) For the high-pressure calibration, proceed as follows. Remove the die assembly, insulation, and vitreous quartz shield. Unscrew both ram extensions. Between the rams, in contact with both of them, position a metal pipe capable of withstanding a 60,000-lbf compression load. Turn on the automatic pressure control. Adjust the Digivider to read 2000 or 3000. From the back of the hydraulic control console, locate trim potentiometer D (Board No. 1) and slowly adjust it until the 4-in. pressure gage reading corresponds to the Digivider setting. (This Step 6 is not obligatory if the gage readings are normal and the machine operation is satisfactory.)

7) To calibrate the electrical pressure signal level, turn on the pressure control system. Set the desired value on the Digivider. Adjust trim potentiometer A (Board No. 1) until you see the desired electrical

voltage-vs-pressure at the signal terminals. (This procedure simultaneously calibrates the pressure recorder.)

8) If the control pressure oscillates—adjust the gain by rotating trim potentiometer B (Board No. 1) until the oscillation just stops.

9) When the calibration has been completed, remove the metal pipe that was positioned in Step 6 and replace all parts that were removed in Step 6.

3.4 Sample-Loading

The procedure followed in loading the sample to prepare it for heating is straightforward but requires meticulous handling of the sample and the equipment. After the die assembly, insulation, and vitreous quartz shield are positioned inside the rf coil, raise the lower ram to its maximum height so that the top face of the bottom punch is positioned at the die center. Place a Grafoil* disk on the punch face. Center the sample in the die cavity. Attach a second Grafoil disk to the upper punch with silicone grease and insert it in the die so that it touches the sample.

Next, lower the upper ram to within 1 in. of the upper punch. To prevent fracturing the sample, avoid premature contact between the ram and punch.

Now position the thermocouple by inserting it through the graphite felt insulation on top of the die body. Place the thermocouple junction in contact with the die body, next to the upper punch.

Close the chamber and evacuate it to the desired vacuum.

The Grafoil serves two functions. First, it is a good lubricant, facilitating deformation and preventing samples from sticking to punch faces. (If samples stick when they cool *in situ*, they may crack because of the differential contraction between samples and punches.) Second, since its density is very nonuniform the Grafoil deforms before the sample does. The resulting slight depressions in both the upper and the lower Grafoil disks hold the sample in place, preventing side slip during initial deformation when the upper and lower sample faces may not be perfectly parallel.

3.5 Sample-Heating

At low temperatures, 300°C or below, vacuum operation is not necessary but is used to retard the oxidation of metal or graphite furnace elements. The temperature of the die cavity is carefully measured as a function of the control thermocouple temperature. Both thermocouples give identical readings when thermal equilibrium is established. A minimum of 1/2 hr is required for equilibration.

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*Union Carbide Corp., 270 Park Ave., New York, N.Y. 10017

Samples are heated slowly over a 2- to 6-hr period, using the Trend Trak curve-follower to program the temperature. Once the forging temperature is achieved, 1 hr is allowed for equilibration, after which forging is begun. The temperature is kept constant until the end of the forging.

3.6 Beginning of Forging

Before forging can actually begin, a threshold must be determined.

Position the upper ram at least 1 in. above the upper punch. Then turn on the automatic pressure control.

Turn the Digivider from zero to small values until upper ram motion just begins. Then turn the Digivider back until ram motion just stops. Repeat several times until the value at which ram motion first begins is consistently the same. This value is the threshold.

Use a continuous dial indicator gage with 0.001-in. increments to observe upper ram motion. The lower ram remains essentially stationary.

Manually lower the upper ram to within about 1/4 in. of the upper punch. Using the automatic pressure control, set the Digivider slightly higher than the threshold value. This will lower the upper ram until it makes contact with the upper punch. When the ram is very close, reduce the Digivider setting to slow the ram descent.

For a threshold in the zero-to-30 range, a Digivider setting at the threshold or one unit above is generally sufficient to make contact. Once contact is made and ram motion stops, reduce the Digivider setting below the threshold to allow final preforging preparations to be accomplished.

If the calibration for the hydraulic control system is stable, the same threshold value will be obtained on every run. This is in fact a shortcut to checking the calibration: merely measure the threshold and compare it with those of previous runs. If it has not changed significantly, recalibration is unnecessary.

An alternative method for determining whether calibration is required is to continually monitor the pressure transducer output. If the zero pressure output from the transducer is unchanged from that of the previous calibration, that calibration is still valid and need not be repeated.

3.7 Stress and Strain

When the Digivider is set at the threshold value and the rams and upper punch are in contact, essentially no force is applied to the sample. Only after the Digivider setting exceeds the threshold is there any force on the sample. The load L on the sample is

$$L = 19.63(D-T),$$

where 19.63 is the ram piston area in inches squared, D is the Digivider setting, T is the threshold. The Digivider dial is calibrated in units of pound-force per square inch.

The forging stress on the sample at any time is calculated by dividing L by the cross-section area at that time. The instantaneous cross section can be found, approximately, by dividing the initial volume of the sample by the instantaneous height.

With the rams in contact with the punches, set the dial indicator gage to zero (zero deformation of the sample). Set the digital timer to zero also. Turn on the timer, set the Digivider to the threshold value, and read the dial indicator. At intervals (usually 1 min apart), gradually increase the Digivider setting, and record the time, Digivider setting, and dial reading. After any time interval Δt (in minutes), the approximate strain rate is given by

$$\text{Strain Rate} \approx \frac{\Delta\,\text{dial}}{\ell} \div \Delta t \quad ,$$

where Δdial is the difference from the previous dial reading, ℓ is the instantaneous sample length, Δt is the interval since the previous reading. The instantaneous sample length is given by:

$$\ell = \ell_o - \text{dial reading} \quad ,$$

where ℓ_o is the initial length of the sample. The dial gage reading represents the total deformation in inches.

It has been our experience that billets of high quality cannot be forged unless the strain rate is increased gradually to optimum value by making small incremental increases in the Digivider setting. Overenthusiastic haste at this point can produce instantaneous strain rates much higher than desired, resulting in high residual strain and sometimes in veiling and cracking in the finished billet.

3.8 Completion of Forging

When the desired strain rate is achieved, continue deformation until the required sample height reduction is reached. To maintain a constant strain rate it may be necessary to vary the size of the Digivider increments, occasionally including zero increments. Increment sizes must be determined through experience with each new material.

After deformation is complete, set the Digivider back to zero, removing the forging stress. Depending on the forging temperature and sample material, either program the temperature down to room temperature or turn the power supply off.

The cooling rate used can significantly affect the residual strain present in forged billets. Cooling rates on the order of 20 C^o/hr or less, as used in annealing the forging blanks, are the most effective in producing forgings of low residual strain.

3.9 Removal of Sample From Hot Press

After the sample has cooled to room temperature, fill the vacuum chamber with air and open it. Raise the upper ram to its highest position by firmly* turning the upper ram position control valve to the RAISE position. Next, remove the upper punch and depress the lower ram to its lowest position. The sample should now be visible just below the die platform (item 20 in Figure 3). Remove the sample gently.

3.10 Clean Up of Sample and Preliminary Evaluation

The Grafoil lubricant usually adheres to the surface of a forged billet. Its nonuniform density creates an orange peel texture on the billet surface, on which the depressions are filled with Grafoil. This is easily removed with room-temperature distilled water and a Kimwipe. Remove all traces of Grafoil. Dry the billet and examine it for cracks, edge tears, veils, and any other patent defects. Then examine it under polarized light to determine qualitatively what residual strains are present.

Polish one surface of the billet to a scratch-free finish. Then etch it in a solution that will make the grain boundaries visible and reveal the microstructure. For KCℓ or doped KCℓ forgings, a 3- to 5-sec dip in room-temperature distilled water, rapidly followed by a rinse in dry methanol and drying in a stream of warm air, is sufficient to reveal the microstructure under a Nomarski phase-interference microscope. Routine microscopic examination of all forgings should be an integral part of the forging procedure. It is very worthwhile, especially when forging a new material, to examine the entire billet surface and evaluate the microstructure to provide a quick input for planning subsequent forging experiments. Later characterization, particularly if conducted by someone extraneous to the experiment, will delay this input and might even be restricted to evaluation of only selected locations on the billets. To determine average grain size from photomicrographs, use the line-intercept method.

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*It is important to turn these valves without hesitation—if the handle is turned slowly, the ram will go down, and downward motion at this point will damage the sample.

4. RESULTS

To date, several different materials have been hot-forged, including KCl, doped KCl, NaCl, and CaF_2; TiO_2 powder has also been hot-pressed. Figure 5 is a photograph of a Czochralski-grown RbCl-doped KCl crystal and a forged billet of the same composition. Figure 6 is a photomicrograph of a RbCl-doped KCl billet.

Table 1 lists the conditions and results of some recent forgings in which a significant increase in mechanical strength was achieved through a more refined materials handling and processing technique. The work is due for publication in the Proceedings of the Fifth Annual Conference on Infrared Laser Window Materials.

Figure 5. Forging Blank and Forged Billet

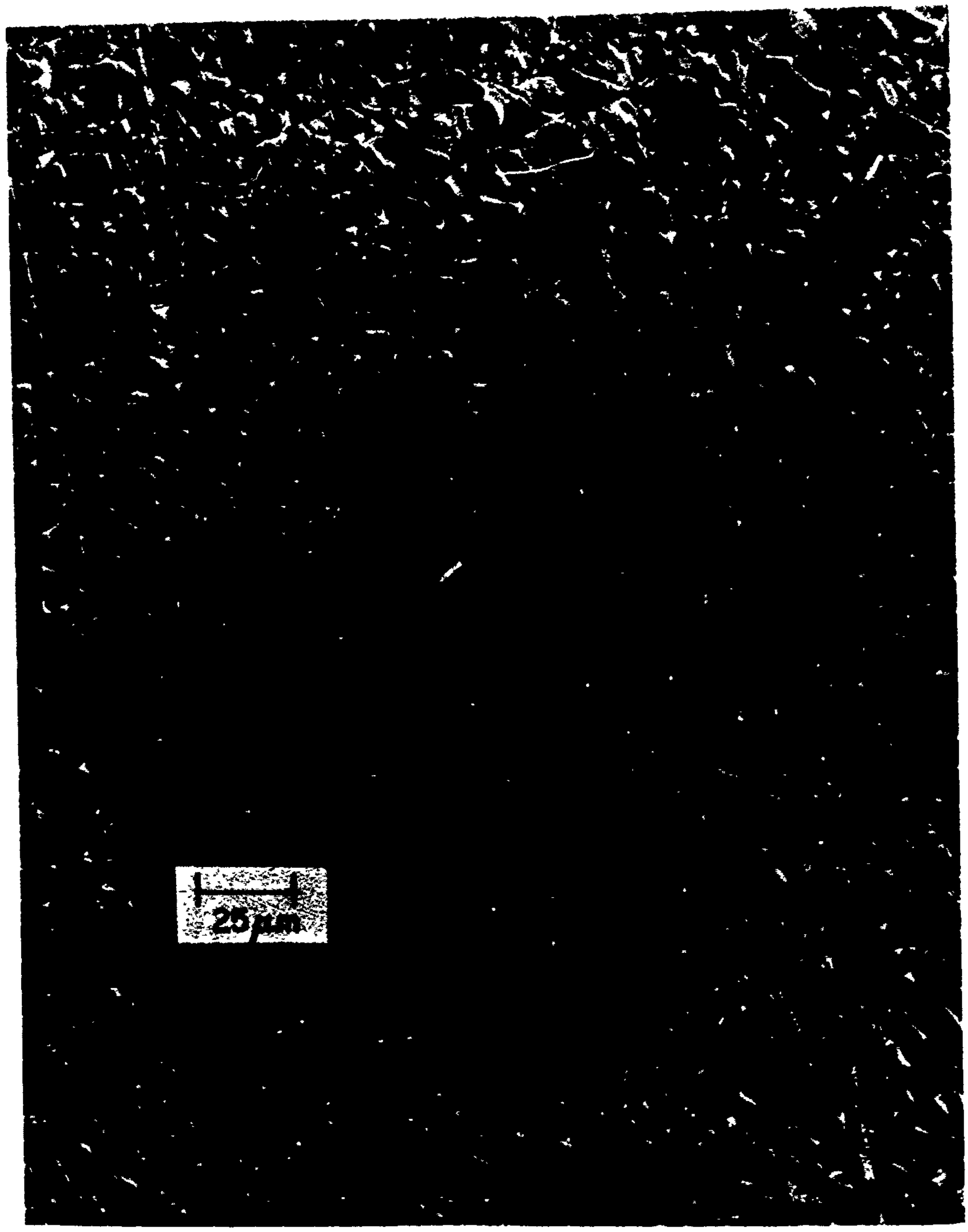

Figure 6. Photomicrograph of KCℓ:RbCℓ

Table 1. Hot-Forging Conditions and Results for KCℓ:RbCℓ

Sample No.	Mole % RbCℓ by Atomic Absorption	Forging Temperature (°C)	Strain Rate (in/in/min)	Final Forging Stress (lbf/in^2)	%Reduction in Sample Height	Grain Size (μm)	Proportional Limit (lbf/in^2)	Rupture Strength (lbf/in^2)
LQ-552	0.957	300	<0.01	1795	55	9.3	7,476	8,985
							7,641	9,476
							8,339	10,595
							7,624 (MIT)	8,934 (MIT)
LQ-553	1.226	300	<0.01	2180	55	7.8	8,652	10,119
							7,657	8,614
							*	6,334
							*	5,376
LQ-554	1.594	300	<0.01	1790	55	8.3	8,816	9,893
							7,515	8,539
							5,637	6,517
							8,552 (MIT)	9,343 (MIT)
LQ-555	2.32	300	<0.01	1640	55	6.2	*	8,500
							8,397	9,169
							10,433	12,520
							* (MIT)	5,418 (MIT)**

*Ruptured without yielding

**This sample was too small for the MIT sample mount; measured value probably low

5. COMMENTS

The empirical results accruing through daily operation of the vacuum hot-pressing system described in this report have endowed AFCRL with a hot-forging capability that is a unique resource in the armamentarium of the U. S. Air Force.

Funding limitations at the time this equipment was procured prevented total automation of all subsystems. At present, the strain rate and ram rate controls are manual, requiring two operators working seven manhours on short runs and thirteen manhours on long runs. Automation will reduce requirements to one operator working two manhours on short runs and three manhours on long runs.

An automated control system has been designed. It offers the benefits of cost (labor) savings and improved performance. When it is procured and installed, its initial advantage will lie in the savings in manpower. And since automated strain rate and ram rate controls are smoother acting than manual controls, the concomitant advantage will be the production of higher-quality forged billets.

Appendix A

Effect of Strain Rate on Sample Quality; Alkali Halides

The vacuum hot-pressing system can be operated under manual control over a wide range of strain rates. The quality of the forging generally improves as the strain rate decreases, subject to the length of the forging run, however, because it is extremely tedious and difficult to maintain control manually for more than 120 to 150 min.

At any fixed forging temperature, the strain rate has a weak effect on grain size but a strong effect on residual strain, veil formation, cracking, and edge-tearing in forged billets. Furthermore, excessive strain rates lead to the formation of unstable microstructures, with subsequent grain growth during cooling or at room temperature. Higher strain rates will produce slightly smaller grain sizes but will also increase the risk of cracking or edge-tearing. Veils are formed at moderate strain rates and can serve as nucleation sites for cracks when strain rates are increased.

It is useful to correlate the microstructures achieved and their stability as well as tendencies toward cracking and edge-tearing with the form of the stress-strain curves used during forging. We define the following:

σ = the forging stress

= L/a (lbf/in^2)

≡ true stress

d = diameter of the billet (in.)

h = height of the billet (in.)

a = cross-section area of the forged billet (in^2)

L = load applied to the sample (lbf)

σ_y = flow stress

μ = coefficient of friction (μ = 0.12 for Grafoil)

Curve I in Figure A1 represents a typical stress-strain curve for KCℓ at 250°C, Grafoil lubricant, and a strain rate on the order of 0.01 in/in/min. After the strain reaches approximately 0.4, a reasonably flat region in the stress-strain curve occurs, corresponding to a wide range of deformation during which the microstructure is fully developed. When the sample impinges on the die wall (see Appendix B), there is a sharp increase in the stress required to continue deformation. As the forging temperature is increased, the shape of Curve I is preserved but the stress level for every strain value is reduced. Also, since frictional effects are reduced at higher temperatures, the slope of segment AB tends toward zero. The same forging run as in Curve I is shown in Curve II except that all

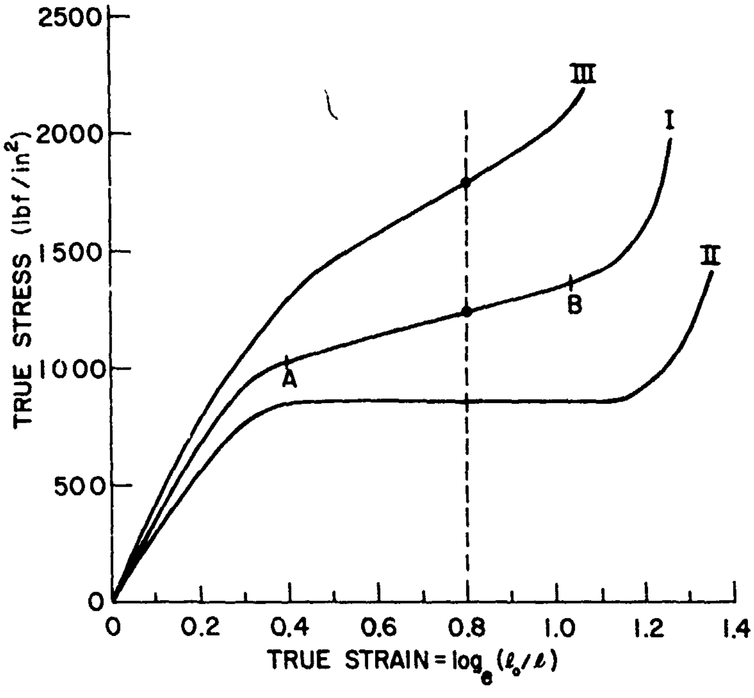

Figure A1. Stress-Strain Curves During Forging of KCℓ at 250°C

frictional effects have been corrected for by using the equation[2]

$$\sigma_y = \frac{\sigma}{1 + \frac{\mu d}{3h}} .$$

Note that from approximately strain = 0.4 to 1.2 the deformation takes place at a constant forging stress.

Curve III shows a forging run carried out under the same conditions as Curve I except that the strain rate is much higher. For any strain value, a significantly higher stress value is required at the higher strain rate. We have observed that forgings following Curve III have nigher residual strain than forgings following Curve II.

The stress-strain curves shown may in practice have some local irregularities caused by fluctuations in the strain rate because of manual rather than automatic control of the strain rate. The slope of the stress-strain curve may even become negative if the strain rate becomes high enough or if a high strain rate persists long enough. First there will be a sharp increase in slope as the strain rate increases; then, when the strain rate is reduced to allow material deformation to catch up with the applied stress, the slope may go negative. Such 'bumps' on the stress-strain curve are highly undesirable because runs where such events occur generally produce inferior forgings.

Curve II is representative of the optimum operating conditions. Forgings must be carried out to strain > 0.4 (point A) to ensure a fully developed microstructure but to strain < 1.0 (point B) to prevent building-in residual strain. Following this procedure has produced the most stable microstructures and the most nearly flawless forged billets.

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2. Yan, M.F., Cannon, R.M., Bowen, H.K., and Coble, R.L. (January 1975) Stabilization of the grain size in hot-forged alkali halides, Proc. Fourth Annual Conference on Infrared Laser Window Materials, pp. 639-665. (Held in Tuscon, Ariz., 18 to 20 November 1974, sponsored by AFML of Wright-Patterson AFB); also AFCRL-TR-75-0170 (AD No. A008480)

Appendix B

Impingement of Samples on Die Wall

The forging technique described in this report allows a fully unconstrained deformation of the forging blank. No constraining rings are used nor is the sample allowed to impinge on the die walls. The only slight constraint that results is from friction between the sample and the punch faces. Consequently, the deformation produces a sample whose shape is determined by the crystallographic orientation of the forging blank.

As indicated in Appendix A, sample impingement on the die wall produces a sharp increase in slope of the stress-strain curve. Furthermore, this produces a sharp increase in residual strain in the forged billet. Figure B1 is a photograph of a billet taken under polarized light; the two upper corners, which had impinged on the die wall, show considerably more strain than the lower corners, which had not. If sample deformation continues beyond initial impingement, the high-residual-strain area increases toward the center of the sample. Any type of constraint, including a confining ring, tends to oppose the natural direction or shape of a deforming crystal and probably introduces residual strain. This effect is more pronounced as total deformation is increased.

The fully unconstrained technique necessitates careful positioning of forging blanks and precise control of total deformation to avoid impingement of samples on die walls. Since the die wall is subjected to essentially zero force, it could be fabricated from low-strength graphite* at a considerable cost saving. Only the

*NOTE: If this technique is used with low-strength graphite, sample impingement on the die body will cause the die to rupture.

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punches would have to be made of high-strength Poco graphite. The die serves only as a susceptor for rf heating and a positioning guide for the punches.

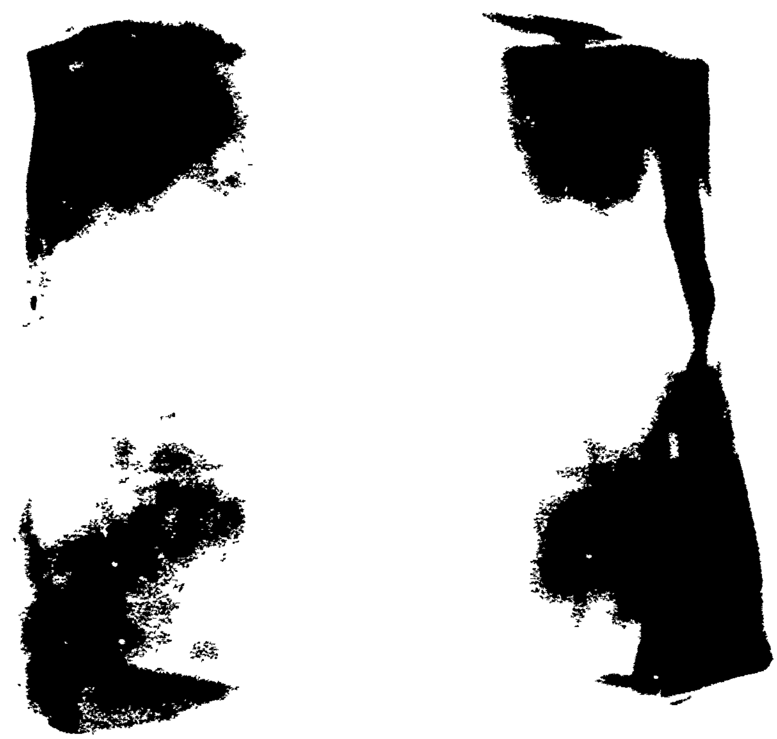

Figure B1. Strain Induced by Impingement on Die Wall